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J. S. Waka and M. H. Riekerung, 'Optics'
 Applications of Chemical Vapor Deposited β -SiC,
 SPIE Proc. Vol CR67, 71-103 (July 1997)

94 / Critical Reviews Vol. CR67

attractive because (i) its thermal expansion coefficient matches quite well with that of SiC over a wide range of operating and deposition conditions, (ii) it can be polished to a very smooth surface, and (iii) it can be diamond turned which permits fabrication of aspheric surfaces in a cost effective manner. Cladding of Si on SiC faceplate can be performed either by a CVD^{54,55} or evaporation process.

Since SiC is a brittle ceramic material, it is susceptible to the flaw induced fracture. The flaw size, C_f in a brittle material is given by the formula⁹:

$$C_f = 0.79 (K_{IC} / \sigma)^2$$

where K_{IC} is the fracture toughness, and σ is the strength of the SiC part. Since the fracture toughness of the material is a constant, the strength of the part depends upon the size of the flaw in the material, which in turn depends upon the volume of the material used in the part. Thus larger is the size of the part, the higher is the probability of finding a flaw of larger size. For SiC with $K_{IC} = 3.4 \text{ MNm}^{-1.5}$, and $\sigma = 421 \text{ MPa}$, the flaw size is about $52 \mu\text{m}$ which is quite small and is a few times the grain size of the material. The maximum allowable stress, σ in large parts can be calculated from the following formula:

$$\sigma = \sigma_1 (A_1/A)^{1/m}$$

where σ_1 is the mean fracture stress for the test specimens, A is the area of the large part, A_1 is the area of the test specimen and m is the Weibull modulus. For SiC, $m = 11.45$, $\sigma_1 = 421 \text{ MPa}$, $A = 160 \text{ mm}^2$. Consequently, for a 1-m diameter part, the allowable maximum stress in the part is 200 MPa. For as-grown SiC surfaces however, the value of m was determined to be about 4 with $\sigma_1 = 262 \text{ MPa}$ ⁵⁶. In this case the allowable maximum stress in the 1-m diameter part is only about 31 MPa which is quite small. These calculations indicate that while fabricating large size mirrors by the CVD process, one should take extra care to ensure that the SiC deposit is not stressed beyond the allowable values during furnace cool-down.

Precision Replication: Precision replication is used when a large number of identical mirrors of CVD-SiC are required⁵⁷⁻⁵⁹. Since in the CVD process, SiC is deposited on the mandrel atom by atom, it is possible to replicate a surface down to the atomic scale. Precision replication is performed by depositing SiC on highly polished mandrels. The candidate mandrel materials are SiC, graphite, sapphire, molybdenum and tungsten⁵⁷. The latter three mandrel materials have thermal expansion coefficient larger than that of SiC. The thermal expansion coefficient of graphite depends upon its grade. Consequently, graphite, sapphire, Mo and W can be used for replicating concave or female parts. For replicating convex or male parts, SiC and graphite are the two preferred mandrel materials. Since graphite cannot be polished to a very smooth surface, it can be clad with a layer of SiC or SiO_2 and the optical surface can be fabricated in the clad layer. Since SiC adheres to SiC, a release coating is required to separate the mandrel from the deposit. Sapphire does not require a release coating and readily separates from the SiC deposit due to a significant thermal expansion mismatch. The other two mandrel materials, Mo and W gets etched in the CVD-SiC process due to the

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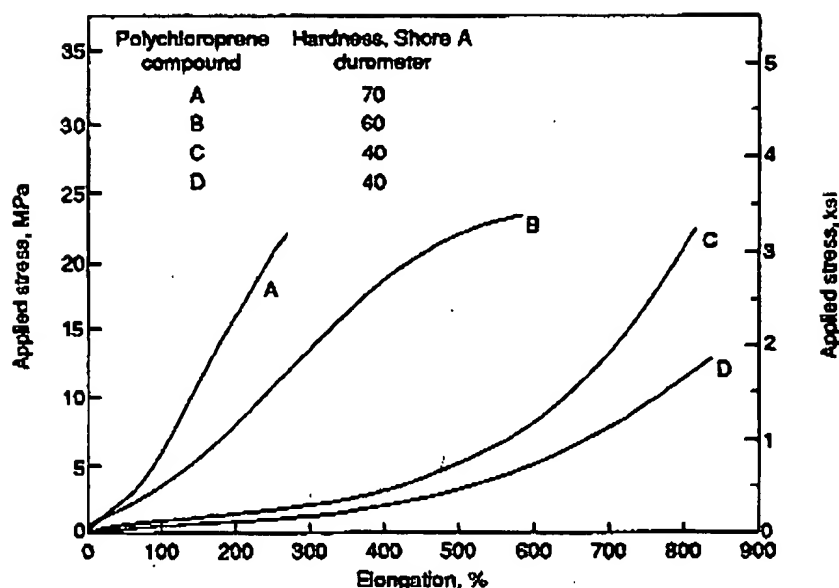


Fig. 25 Tensile-test curves for four polychloroprene compounds

chine. The filaments are then stressed to failure at a constant strain rate. For this test method, filament cross-sectional areas are determined by planimeter measurements of a representative number of filament cross sections as displayed on highly magnified micrographs. Alternative methods of area determination use optical gages, an image-splitting microscope, a linear weight-density method, and others.

Tensile strength and Young's modulus of elasticity are calculated from the load elongation records and the cross-sectional area measurements.

The specimen setup is shown in Fig. 26. Note that a system compliance adjustment may be necessary for single-filament tensile modulus.

Tow Tensile Test (ASTM D 4018). The strength of fibers is rarely determined by testing single filaments and obtaining a numerical average of their strength values. Usually, a bundle or yarn of such fibers is impregnated with a polymer and loaded to failure. The average fiber strength is then defined by the maximum load divided by the cross-sectional area of the fibers alone.

Using ASTM D 4018 or an equivalent is recommended. This is summarized as finding the

tensile properties of continuous filament carbon and graphite yarns, strands, rovings, and tows by the tensile loading to failure of the resin-impregnated fiber forms. This technique loses accuracy as the filament count increases. Strain and Young's modulus are measured by extensometer.

The purpose of using impregnating resin is to provide the fiber forms, when cured, with enough mechanical strength to produce a rigid test specimen capable of sustaining uniform loading of the individual filaments in the specimen.

To minimize the effect of the impregnating resin on the tensile properties of the fiber forms, the resin should be compatible with the fiber, the resin content in the cured specimen should be limited to the minimum amount required to produce a useful test specimen, the individual filaments of the fiber forms should be well collimated, and the strain capability of the resin should be significantly greater than the strain capability of the filaments.

ASTM D 4018 Method I test specimens require a special cast-resin end tab and grip design to prevent grip slippage under high loads. Alternative methods of specimen mounting to end tabs are acceptable, provided that test specimens maintain axial alignment on the test machine centerline and that they do not slip in the grips at high loads. ASTM D 4018 Method II test specimens require no special gripping mechanisms. Standard rubber-faced jaws should be adequate.

Mechanical Testing of Ceramics

Ceramic materials have been used in a variety of engineering applications that utilize their wear resistance, refractoriness, hardness, and high compression strength. Traditionally, they have not been used in tensile-loaded structures because they are brittle and experience catastrophic failure before permanent deformation. Nevertheless, their extreme refractoriness, chemical inertness, and favorable optical, electrical, and thermal properties are inducements to use ceramics in certain tensile load-bearing applications. Typical mechanical properties of common ceramics are listed in Table 11, and applicable ASTM standards for mechanical testing are listed in Table 12. More current information on mechanical testing of ceramics is provided in Ref 22.

Room-Temperature Strength Tests

Uniaxial Tensile Strength. The nonductile nature of monolithic ceramics and their high sensitivity to stress concentrators has meant that conventional direct tensile testing is difficult and expensive. Gripping with jaws, screw threads, or other conventional devices causes invalid test results because of specimen breakage at the grips. The high stiffness (elastic modulus) of many ceramics means that a misalignment

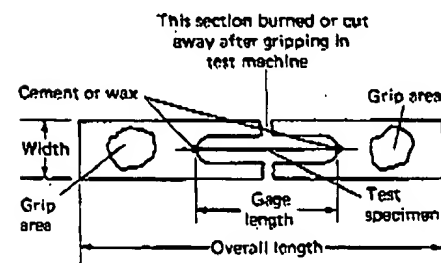


Fig. 26 Schematic showing typical specimen-mounting method for the single-filament fiber tension test (ASTM D 3379)

Table 11 Typical mechanical properties of common ceramic materials

Material	Young's modulus		Flexural strength		Compressive strength	
	GPa	10 ⁶ psi	MPa	ksi	MPa	ksi
Brick	3-20	0.7-2.9	5-10	0.7-1.5	10-25	1.5-3.6
Roof tile	5-20	0.7-2.9	8-15	1.2-2.2	10-25	1.5-3.6
Steatite	1-3	0.1-0.4	140-160	20-23	850-1000	123-145
Silica refractories, 96-97% SiO ₂	8-14	1.2-2.0	30-80	4.4-11.6
Fireclay refractories, 10-44% Al ₂ O ₃	20-45	2.9-6.5	5-15	0.7-2.2	10-80	1.5-11.6
Corundum refractories, 75-90% Al ₂ O ₃	30-120	4.4-17.4	10-150	1.5-22	40-200	5.8-30.7
Forsterite refractories	25-30	3.6-4.4	5-10	0.7-1.5	20-40	2.9-5.8
Magnesia refractories	30-35	4.4-5.1	8-200	1.2-29	40-100	5.8-14.5
Zircon refractories	35-40	5.1-5.8	80-200	12-29	30-60	4.4-8.7
Whiteware	10-20	1.5-2.9	20-25	2.9-3.6	30-40	4.4-5.8
Stoneware	30-70	4.4-10.2	20-40	2.9-5.8	40-100	5.8-14.5
Electrical porcelain	55-100	8.0-14.5	90-145	13-21	55-100	8.0-14.5
Capacitor ceramics	90-160	13-23	300-1000	44-145

Source: Ref 21

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of only a few thousandths of a centimeter can lead to bending stresses with errors of 10% or more. Specimen preparation to exacting tolerances with minimal machining damage and careful tapers to avoid stress concentrators has been an expensive proposition. Considerable work has focused on improving tensile test methods for ceramics, with the result that tensile testing is becoming more routine. Commercial equipment is readily available, and specimen costs are falling. It will, however, always be more difficult to conduct direct tensile tests for ceramics than for metals.

The experimental difficulties, coupled with the problems of fabricating sufficiently large specimens, have prompted ceramists to use alternative test methods. The most common is flexure testing, in either the so-called three-point or four-point configuration. The latter is usually further specified by a description of the distance from the outer support points and the inner points, such as $\frac{1}{4}$ or $\frac{1}{2}$ four-point loading. The small size, low cost, and easy preparation of a flexure specimen account for its popularity, but there are distinct drawbacks. The bending creates a stress gradient in the specimen, and only a small volume is exposed to high tensile stress. The specimens are very sensitive to edge or surface machining damage. The test appears easy to set up and conduct, but misalignments and experimental errors can easily ruin it. Standard test methods are now available that

permit accurate strength measurements for standard sizes and shapes, as shown in Fig. 27.

Nevertheless, it is still preferable to perform direct tension testing. Current testing systems are designed with self-aligning features that limit the imposed bending stresses to approximately 1%. There is usually less extrapolation of the strength data from test specimen to component size. Tensile specimens are still expensive, however, because of costly fabrication and machining. They are inconveniently large, as well, because most systems are designed for high-temperature test rigs that use cold grips. Until recently, only a few laboratories had the ability to test or to even afford direct tensile experiments. A new emphasis on attaining accurate, quality data in support of ceramics in heat engine programs has led to rapid improvements in the field, and commercial test systems are now readily available. Different tensile specimen geometries that are being used are shown in Fig. 28 (Ref 23-28).

Another occasionally cited test for engineering ceramics is the so-called diametral compression test, or Brazilian disk test, wherein a circular cylinder is loaded at its ends (Ref 29, 30). The test is actually biaxial, because in addition to the tensile stresses that tend to laterally split the specimen, compressive stresses that are three times as great act axially through the specimen. However, compressive stresses of this magnitude are not likely to affect uniax-

ial strength, an effect peculiar to monolithic ceramics. The specimen loading is between two platens with pads of compliant material (such as a metallic shim or paper) to avoid high shearing stresses. Careful machining of the end faces of the specimen is essential, once again to avoid damage that compromises the test. This point is often overlooked. This test is occasionally employed by ceramics processors for ceramics fabricated in cylindrical shapes.

Many ceramic materials have strengths that are specific to the shaping process being used, such as injection-molded turbocharger rotors or extruded heat-exchanger tubes. In such cases, it is not practical to cut tensile specimens from the part, but separately cast tensile specimens may not have the same microstructure or defects as the component and, therefore, are irrelevant (Ref 31-33). It is optimal to test components in as close a configuration to the final component shape as possible. Thus, in the case of a tube, a ring can be cut from the tube and pressurized to obtain a uniaxial hoop-stress-testing configuration (Ref 34, 35). Contrary to expectations, such a test can be conducted at high temperatures. Indeed, one of the highest recorded strength test temperatures for a ceramic (2180 °C, or 3955 °F) was on a pressurized tube (Ref 36). Extreme care must be taken to ensure that the edges are not chipped and do not have excessive machining damage, lest the test merely become a measure of machining damage.

There is no simple answer to the question of what specimen is best for measuring strength data. The best practice is to test a configuration that most resembles the actual component in its service conditions and to ensure that the test material accurately represents the component material. It is likely that the first available data will be flexure-strength data, which are typically higher (10-50%) than tensile specimen data because of the dependency of strength on test specimen size. Nevertheless, considering the tradeoffs in cost, quantity of results, and difficulty in testing, it is likely that future engineering databases will feature complementary flexure and tensile data. Indeed, it will be beneficial to have strength data from different sizes and shapes to permit an assessment of material consistency, flaw uniformity, and the veracity of strength-size scaling models.

Elastic Modulus. Several methods are used to evaluate the elastic moduli of monolithic or fine-scaled, isotropic composites. The most common are deflection measurements in flexural strength tests (with proper consideration of the test machine compliance) or strain gage experiments in flexure or direct tension. Dynamic measurements are also quite common, with either sonic excitation of prismatic specimens at their resonant frequency or time-of-flight measurements of ultrasonic waves.

Interpretation of Uniaxial Strength. The scatter in uniaxial strengths is well modeled by Weibull statistics. Weibull observed that the strength of brittle materials is controlled by the presence of randomly distributed defects and

Table 12 ASTM standards related to mechanical testing of ceramics

Terminology	
C 1145	Standard Definition of Terms Relating to Advanced Ceramics
C 1286	Standard System for Classification of Advanced Ceramics
Properties and performance (monolithic)	
C 1161	Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
C 1211	Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures
C 1259	Standard Test Method for Dynamic Young's Modulus
C 1273	Standard Practice for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperature
Design and evaluation	
C 1175	Standard Guide to Test Methods for Nondestructive Testing of Advanced Ceramics
C 1198	Standard Test Method for Dynamic Young's Modulus
C 1212	Standard Practice for Fabricating Ceramic Reference Specimens Containing Seeded Voids
C 1239	Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
Characterization and processing	
C 1251	Standard Guide for Determination of Specific Surface Area of Advanced Ceramics by Gas Adsorption
C 1274	Standard Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption
C 1282	Standard Test Method for Determination of the Particle Size Distribution of Advanced Ceramics by Centrifugal Photosedimentation
Ceramic composites	
C 1275	Standard Practice for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Section at Ambient Temperatures

Source: Ref 21

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that failure is controlled by the largest, most severely stressed defect. Fracture occurs when a defect in one particular element of the body reaches a critical loading. This analysis is colloquially known as the weakest-link model, in direct analogy to the strength of a chain.

The Weibull modulus, m , has no units and is the factor that determines the scatter in strength. High values are optimum. Traditional ceramics, such as whitewares and brick, may have values from 3 to 5. A good material has a value that exceeds 10. A ceramic with an m value ≥ 30 has very consistent strengths and could be practically considered to have a deterministic value of strength over a range of several orders of magnitude volume.

Not only does strength scale with specimen size, but the magnitude of the change strongly depends on whether the defects are surface or volume. Obviously, it is essential to know whether flaws are of one or the other category if the laboratory strength data are going to be size-scaled to predict component performance.

A Weibull graph is a convenient means to report strength data. The graph usually has special axes chosen to linearize the data. This is done in the same fashion that probability paper

can be used to linearize data for a Gaussian distribution.

The Weibull analysis is adequate for multiaxially, tensile loaded ceramics, provided that the second or third principal stresses are significantly less than the principal tensile stress. If this is not the case, then it is appropriate to use more sophisticated analyses that take into account the effect of multiaxial tensile stresses on defects. The Weibull analysis also has limitations if the defects are likely to grow subcritically during a test. A newly recognized phenomenon that could occasionally pose problems in strength analysis is *latent* defect caused by localized surface impact or contact stresses. Concentrated microdamage can occur that can lead to a larger microcrack popping in after an incubation period (Ref 37, 38).

Strength values by themselves are only half the picture. The types of defects are equally important because each flaw type has its own Weibull distribution, and because multiple flaw populations are common in ceramics. Therefore, it is essential that the defects be as clearly associated with the strength values as possible.

Uniaxial Compression Strength. The high compressive strength of ceramics is a consequence of the resistance of the material to plas-

tic flow and the insensitivity of defects to compressive stress. Ancient structural applications of ceramics were columns and walls that capitalized on high compressive strength. The fact that ceramics fail at all in compression is a result of the distortion of the stress field in the immediate vicinity of the tip of a defect. This distortion causes a localized tensile stress concentration that, for defects at the worst orientation ($\sim 30^\circ$ to the axial stress) is about $\frac{1}{4}$ of the concentration if the specimen is loaded in tension. Thus, a Griffith-type criterion for failure would predict that the compression specimen will fail at about 30° to the specimen axial direction when the compressive stress is eight times the tension strength, but this is an oversimplification.

The tensile stresses in the immediate vicinity of a defect will cause a crack to propagate stably for a slight distance (Ref 39, 40). The crack then aligns itself with the compression stress and is arrested. Progressively more defects grow until the damage that has accumulated in the specimen reaches some limit, and the specimen virulently disintegrates into powder (often with a triboluminescent emission) (Ref 41, 42). Compression strength thus depends not on the largest, worst-oriented, highest-

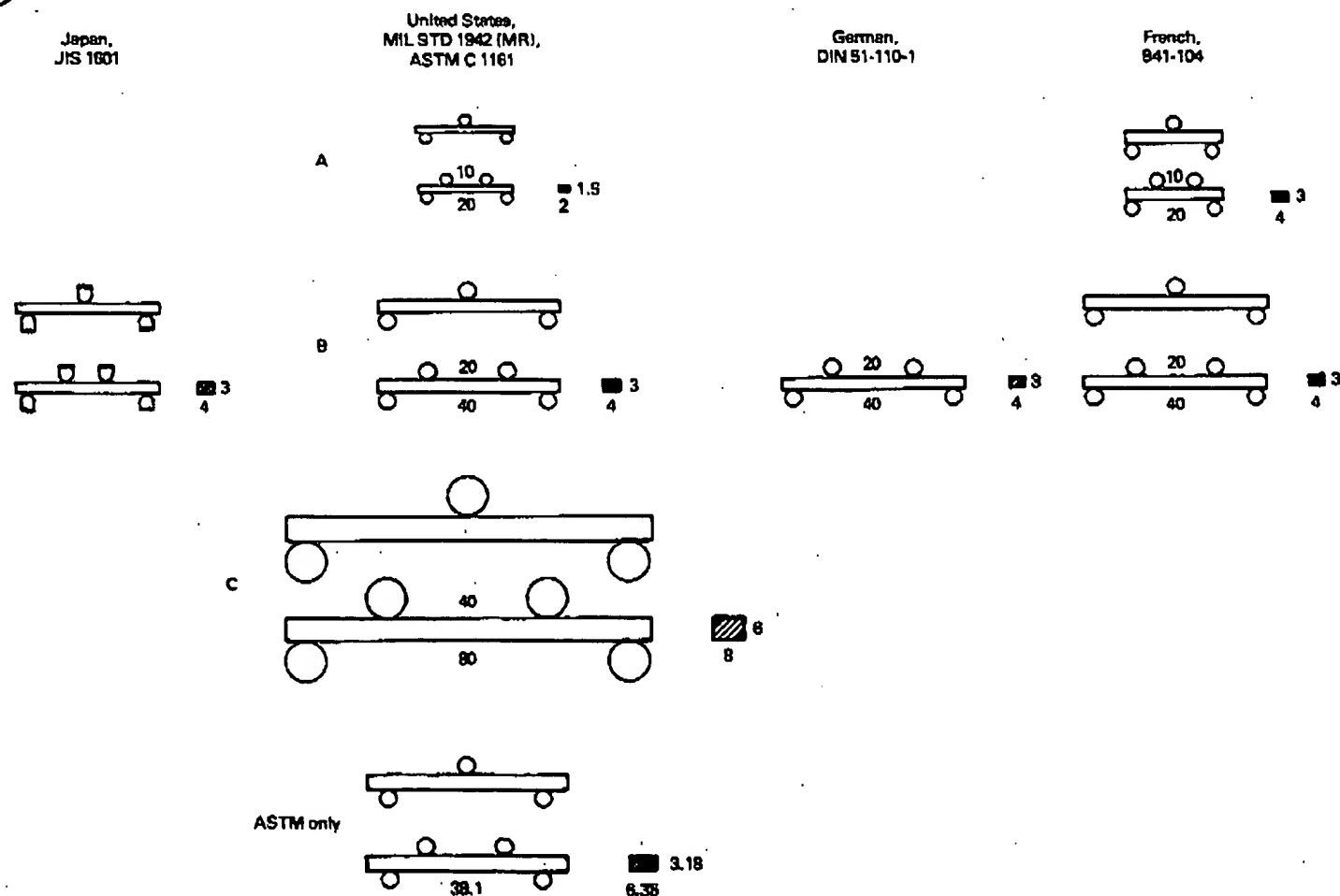


Fig. 27 Flexure strength standard test methods; all dimensions in mm

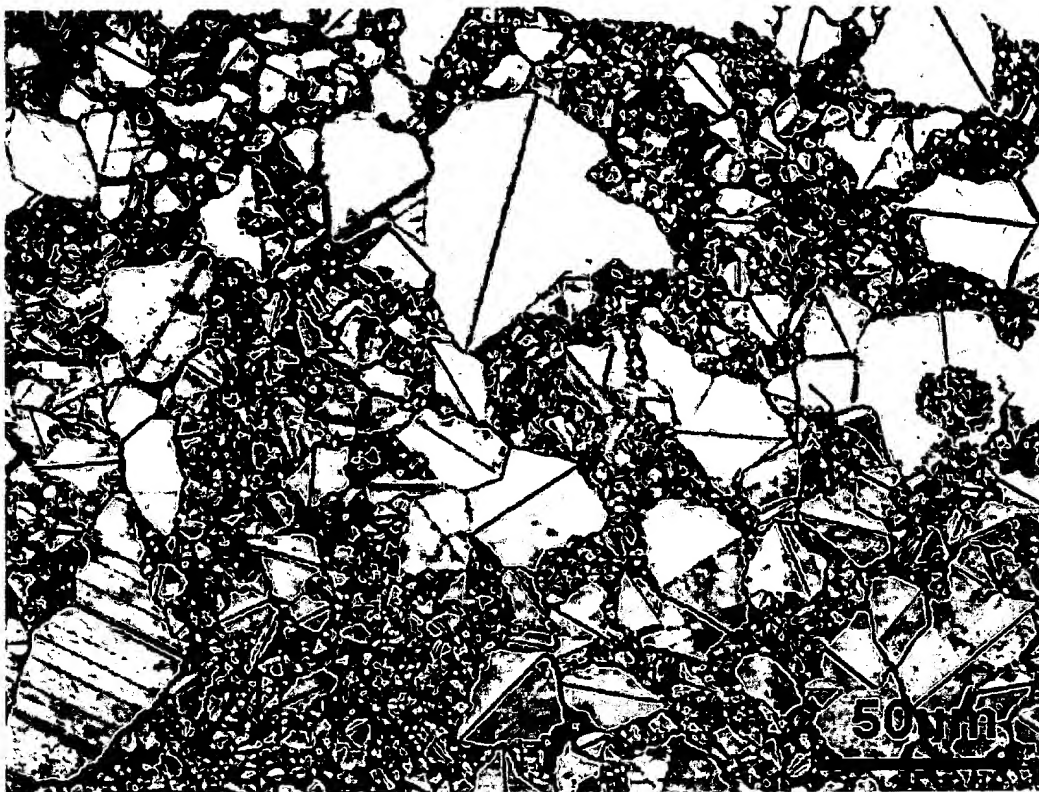


Figure 1: Typical microstructure of CVD-SiC perpendicular to growth direction showing different grains .